# Dyeing of Silk Fabric with Reactive Dye using Polyacrylamide as Exhausting Agent

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***Abstract***

*In this study, Mulberry silk fabric samples were successfully dyed with reactive dye for 0.5, 1, 2, 3 and 4 shade% using polyacrylamide as exhausting agent. Same Mulberry silk fabric samples were dyed with same reactive dye for same shade% using common salt as exhausting agent. Comparison between fabric samples of corresponding shade% showed that the samples dyed with polyacrylamide exhaustion method are slightly lighter in color. Washing and rubbing fastness tests showed that the fastness of both type of samples are very good and they are quite comparable to each other.*

**Keywords** - *Silk Fabric, Reactive Dye, Polyacrylamide, Common salt, Spectrophotometry, Color Fastness*

## I. INTRODUCTION

Silk is one of the most popular protein fiber extruded from silkworm. The importance of silk as a textile fiber lies in its luster, handle and draping qualities. In the early decades of synthetic dyes, silk was dyed mainly with cationic (basic) dyes and only later with anionic dyes, i.e. with acid and direct dyes. Acid dyes tend to give low depth on silk, and if very deep colors are required 'bronzing' occurs. They do not have the wide range of bright colors generally required for silk, so some old basic dyes are still sued for application on silk in spite of their low wash and light fastnesses. The majority of dyeing with acid, basic and direct dyes is poor in wash fastness due to binding forces based on non-polar electrostatic, van der Waals and hydrogen bond interactions only [ l ]. In order to improve the color fastness of silk, recently more than a few attempts have been made at coloration of silk with reactive dyes [2]-[8].

However, like cotton dyeing with reactive dye, large amount of salts are also required as exhausting agent in case of silk dyeing because the carboxylic acid groups of silk gain anionic charges in water which repulse the anionic reactive dyes. The salts remain in the discharged dye liquor which creates enormous environmental problems.

Many researchers in different countries worked to minimize or eliminate the use of electrolyte concentration in reactive dye liquor. For cotton, cationic sites are introduced within the cellulose to increase the dye absorption. For example, quaternary cationic agents having various reactive groups (e.g. epoxy and chlorotriazine) are used to cationize cellulose [9]-[11]. Other fiber-reactive substituted amino compounds are also used for cationization[12]-[14]. Cationization of cotton using polyacrylamide is also reported in different articles [15]-[16]. Use of polyacrylamide as exhausting agent may be a technique to introduce cationic site within the silk polymer structure also. The cationic charged amino groups of polyacrylamide may be involved in absorption of anionic reactive dyes by electrostatic attraction, thus offer a possibility of reactive dyeing of silk without using electrolytes. For this reason, we performed a series of experiments to see the dyeing possibility of silk with reactive dye using polyacrylamide as exhausting agent for different shade%. The results are also compared with silk dyeing process with reactive dye using common salt as exhausting agent.

## II. MATERIALS & METHODS

### *Degumming of silk*

Degumming of required amount of Mulberry silk fabrics is done with recipe shown in table I.

**TABLE I**
**The recipe of silk fabric degumming**

| **Chemicals** | **Amount** |
|---|---|
| Soda ash | 2 g/L |
| Detergent | 2 g/L |
| Temperature | 85 ºC |
| pH | 10.5 |
| Time | 40 min. |
| M:L | 1:30 |

The fabric samples were then thoroughly washed and dried.

**TABLE II**
**The recipe of dyeing of silk fabric with polyacrylamide exhaustion method**

| **Shade%** | **0.5** | **1.0** | **2.0** | **3.0** | **4.0** |
|---|---|---|---|---|---|
| Polyacrylamide % | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 |
| Sequestering Agent (g/L) | 1 | 1 | 1 | 1 | 1 |
| Leveling Agent (g/L) | 1 | 1 | 1 | 1 | 1 |
| Wetting Agent (g/L) | 1 | 1 | 1 | 1 | 1 |
| Soda ash (g/L) | 7.5 | 10 | 12.5 | 15 | 17.5 |
| M:L | 1:30 | 1:30 | 1:30 | 1:30 | 1:30 |
| Fixation Temperature (ºC) | 60 | 60 | 60 | 60 | 60 |
| Time (min.) (Exhaustion + Fixation) | 30+60 | 30+60 | 30+60 | 30+60 | 30+60 |

***Dyeing of silk***

Five samples of silk fabric were dyed with 0.5%, 1%, 2%, 3% and 4% reactive dye (Remazol Red RR - DyStar). 0.1% polyacrylamide was used as exhausting agent. From now this dyeing process will be illustrated as polyacrylamide exhaustion method. The dyeing recipe is shown in table II.

The dyeing process was as follows: Required amount of water was taken in the dye pots of Laboratory dyeing machine (ECO DYER Rapid, China). 0.5% stock solution of polyacrylamide in water was made in a conical flask. Required amount of polyacrylamide solution was taken in the dye pots. 1 g silk fabric samples were taken in each dye pots and soaked the samples for 30 minutes. Then required amount of sequestering agent, leveling agent, wetting agent and anticreasing agent were taken and stirred. 1% stock solution of reactive dye was made in a conical flask. Required amounts of stock solution for each shade% were taken in the dye pots. The samples were agitated at room temperature for 30 minutes for exhaustion. Then the required amount of soda ash was added to each pot and the temperature was raised to 60ºC and dyed for 60 minutes. After dyeing, the samples were thoroughly washed and dried.

**TABLE III**
**Amount of common salt taken in salt exhaustion method**

| **Shade%** | **CommonSalt (g/L)** |
|---|---|
| 0.5 | 30 |
| 1.0 | 40 |
| 2.0 | 50 |
| 3.0 | 60 |
| 4.0 | 70 |

To compare the shade variation of silk fabric samples dyed with polyacrylamide exhaustion method with conventionally dyed fabric samples using common salt (from now illustrated as common salt exhaustion method), five samples were again dyed with same reactive dye for same shade% of 0.5, 1, 2, 3 and 4. The sequestering agent, leveling agent, wetting agent, soda ash, M:L, exhaustion and fixation temperature and required time were kept same as for polyacrylamide exhaustion method. The amounts of common salt taken are shown in table III.

***Spectrophotometry***

X-Rite Spectrophotometer (USA) was used to compare the color differences between the fabric samples dyed with polyacrylamide exhaustion method and common salt exhaustion method.

## III. RESULTS & DISCUSSION

Table IV shows the spectrophotometric results of 0.5%, 1%, 2%, 3% and 4% shade of fabric samples. For all shade%, the fabric samples dyed with common salt exhaustion method are taken as standard. The positive DL* values calculated from CIE 1976 L*a*b* (CIELAB) coordinates (CIE for Commission International de I'Eclairage or International Commission on Illumination, L* the lightness coordinate, a* the red/green coordinate, b* the yellow/blue coordinate) indicate that all fabric samples dyed with polyacrylamide exhaustion method are slightly lighter than their respective standards. The CMC (Color Measurement Committee of The Society of Dyers and Colorists, Great Britain) overall color difference, CMC DE values between common salt exhaustion method and polyacrylamide exhaustion method also indicate that the fabric samples dyed with polyacrylamide exhaustion method are slightly lighter than their respective standards (shown in Fig. 1).

Figure 2 shows the variation of color strength, K/S values with the variation of shade % for both common salt exhaustion method and polyacrylamide exhaustion method. The spectrophotometer determined the K/S values of

**TABLE IV**
**Spectrophotometric results of different shade%**

| Shade% | Illuminant | Lightness difference, DL* | Red/green difference, Da* | Yellow/blue difference, Db* | Chroma difference, DC* | Hue difference, DH* | CMC overall color difference, DE |
|---|---|---|---|---|---|---|---|
| 0.5 | D65 | 6.56 | -3.67 | -0.32 | -3.50 | -1.16 | 3.11 |
| 1.0 | D65 | 5.71 | -3.20 | -1.78 | -2.84 | -2.31 | 2.95 |
| 2.0 | D65 | 5.46 | -1.37 | -1.44 | -1.19 | -1.60 | 2.63 |
| 3.0 | D65 | 7.08 | 0.52 | -1.65 | 0.68 | -1.59 | 3.46 |
| 4.0 | D65 | 5.65 | 2.09 | -1.71 | 2.20 | -1.57 | 2.98 |
| 0.5 | F02 | 6.59 | -2.80 | -0.29 | -2.59 | -1.11 | 3.00 |
| 1.0 | F02 | 6.00 | -2.19 | -1.37 | -1.78 | -1.87 | 2.89 |
| 2.0 | F02 | 5.92 | -0.76 | -0.81 | -0.58 | -0.95 | 2.73 |
| 3.0 | F02 | 7.67 | 0.76 | -0.80 | 0.89 | -0.65 | 3.68 |
| 4.0 | F02 | 6.13 | 1.92 | -1.15 | 2.06 | -0.86 | 3.16 |

fabric samples through Kubelka-Munk equation: $\frac{K}{S} = \frac{(1-R)^2}{2R}$ where R = reflectance percentage, K = absorption co-efficient and S = scattering co-efficient of dyes. When reflectance is less, absorbance is more, indicates more K/S value. The maximum K/S values found at 530 nm wave length are plotted against different shade% for all type of samples (in Figure 2). For all shade%, higher K/S values are found for samples dyed with common salt exhaustion method. However, K/S values found for both exhaustion methods are very closer at lower shade% region.

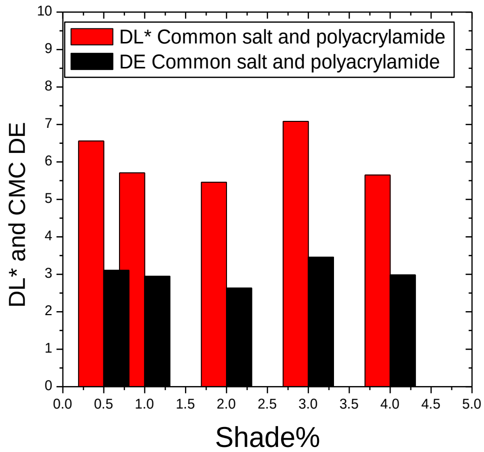


**Fig 1: Variation of DL* and CMC DE values with the increase of shade%**

The amine ($-NH_2$) group of polyacrylamide may be formed protonated amino group when come contact with the carboxylic acid group of silk in aqueous medium as shown in Figure 3, thus attract the negative ions of dye molecules and formed covalent bond with silk in an alkali medium at a temperature 60 °C (as shown in Figure 4). As the polyacrylamide molecules are much larger in size which cannot reach close to all carboxylic acid groups in amorphous region of silk (like the sodium cation of sodium chloride molecules when exhaustion is carried out using common salt in conventional reactive dyeing), the bonded dye molecules with silk may be lower in number, thus the fabric samples dyed with polyacrylamide exhaustion method appeared lighter.

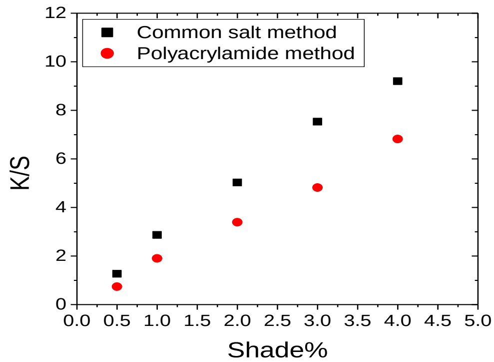


**Fig 2: Increase of color strength (K/S) with the increase of shade%**

***Color Fastness to Wash***

Table V shows the comparison of color fastness to wash between the samples dyed with polyacrylamide exhaustion method and common salt

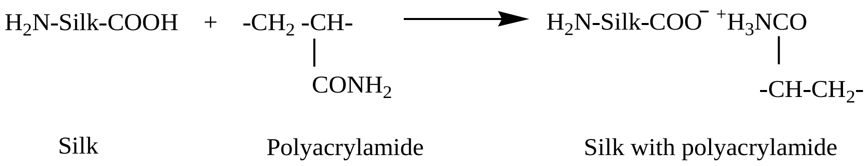


**Fig 3 : Reaction between silk and polyacrylamide**

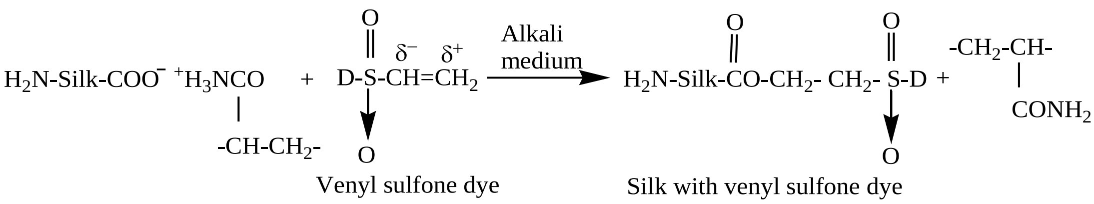


**Fig 4 : Reaction possibilities between polyacrylamide added silk and venylsulfone dyes**

exhaustion method. ISO 105 C60:1994 method is followed to evaluate the wash fastness. This test (C2S) is designed to determine the color which may be transferred from the surface of a colored textile material to multifiber test cloth for washing at 60 $^0$C for 30 minutes. The samples dyed with 3 and 4 shade% have been evaluated for wash fastness test. The sample size was 10 cm x 4 cm and washed with 4 g/L ECE (European colour fastness establishment) reference detergent & 1 g/L sodium perborate solution. For samples dyed with common salt exhaustion method, the grey scale shows rating 5 for both 3 and 4 shade% samples after wash. For samples dyed with polyacrylamide exhaustion method, the grey scale rating is found 4-5 for both 3 and 4 shade% after wash. The multifiber test cloth is consisted of different types of fibers such as di-acetate, cotton, polyamide, polyester, acrylic and wool. The staining of colors on the fibers from the dyed samples has also been evaluated. The results of grey scale rating are almost same for both types of samples as shown in table V.

***Color fastness to Rubbing***

Table VI shows the comparison of color fastness to rubbing between the fabric samples dyed with polyacrylamide exhaustion method and common salt exhaustion method. This test is designed to determine the transfer of color from the dyed samples to the crockmeter test cloth for rubbing. Here dry and wet rubbing fastnesses are compared. ISO 105x12:1993 method is followed to measure the rubbing fastness. The samples dyed with 3 and 4 shade% have been evaluated for rubbing fastness test. The sample size was 14 cm x 5 cm. The dry and wet rubbing fastnesses are found very good with the grey scale rating 5 and 4-5 respectively for both 3 and 4 shade%.

## IV. CONCLUSIONS

Silk fabric samples were successfully dyed with reactive dye using polyacrylamide as exhausting agent. Although comparison between fabric samples of corresponding shade% dyed with polyacrylamide

**TABLE V**
**Color fastness to wash**

| | **For samples dyed with common salt exhaustion method** | | **For samples dyed with polyacrylamide exhaustion method** | |
|---|---|---|---|---|
| | **3% shade** | **4% shade** | **3% shade** | **4% shade** |
| Change in Color | 5 | 5 | 4-5 | 4-5 |
| Di-Acetate (Stain) | 5 | 5 | 4-5 | 4-5 |
| Cotton (Stain) | 4-5 | 4-5 | 4-5 | 4-5 |
| Polyamide (Stain) | 4-5 | 4-5 | 5 | 5 |
| Polyester (Stain) | 5 | 5 | 5 | 5 |
| Acrylic (Stain) | 4-5 | 4-5 | 4-5 | 4-5 |
| Wool (Stain) | 4-5 | 4-5 | 4-5 | 4-5 |

**TABLE VI**
**Color fastness to rubbing**

| Shade% | For samples dyed with common salt exhaustion method | | For samples dyed with polyacrylamide exhaustion method | |
|---|---|---|---|---|
| | Dry | Wet | Dry | Wet |
| 3 | 5 | 4-5 | 5 | 4-5 |
| 4 | 5 | 4-5 | 5 | 4-5 |

exhaustion method and common salt exhaustion method showed that fabric samples dyed with polyacrylamide exhaustion method are slightly lighter but the differences are very small specially at the lower shade% region. Thus silk can be readily dyed with reactive dye using polyacrylamide as exhausting agent without using salts because salts are creating huge environmental problems now-a-days. The wash fastness and rubbing fastness for both types of samples are found very well and they are found quite comparable to each other.

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